

# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

### NO DRAWINGS

#### Set-Retarded Calcium Sulphate Hemihydrate

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED of Imperial Chemical House, Millbank, London S.W.1., a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to set-retarded calcium sulphate hemihydrate compositions.

In many of the applications of calcium sulphate hemihydrate it is advantageous to add thereto an ingredient to retard its setting. For example, wall plasters containing calcium sulphate hemihydrate without a retarding ingredient will set too quickly after mixing with water to give the plasterer adequate time to work the wet plaster on the wall.

Retarding ingredients commonly used include proteinous materials and salts of citric, gluconic and aconitic acid. It is also known that the incorporation of a water-soluble ionic cellulose ether such as sodium carboxymethyl cellulose with calcium sulphate hemihydrate retards the setting of the hemihydrate. In some cases this is a desirable method of retarding the set since the incorporation of a water-soluble cellulose ether into a plaster improves the water-retaining properties of the plaster and facilitates its application. When set-retarded calcium sulphate hemihydrate containing ionic cellulose ethers is used in plaster mixtures containing hydrated lime, the lime reacts with the cellulose ether to form an insoluble calcium salt and the set-retarding effect is not displayed.

For plaster mixtures containing lime it is common to use a conventional set-retarding ingredient and to employ a non-ionic cellulose ether to improve the plaster in other respects.

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We have now found that set-retarding of calcium sulphate hemihydrate may be effected by using as set-retarding agents certain mixed cellulose ethers containing both ionic and non-ionic substituent groups in the cellulose chain.

A set-retarded calcium sulphate hemihydrate according to the invention comprises, as set-retardant, a water-soluble cellulose ether containing both ionic and non-ionic etherifying substituent groups in the cellulose chain, the degree of substitution of the ether by the ionic groups being such that the ether does not form an insoluble calcium salt in the presence of a saturated solution of calcium hydroxide.

The ionic substituent groups in the cellulose ether may conveniently be sodium carboxymethyl and the non-ionic groups may be alkyl, hydroxyalkyl or alkylhydroxyalkyl.

The cellulose ether is preferably sufficiently fine for substantially all of it to pass a 50 mesh B.S. sieve and should have a high viscosity. It is preferred to use material of viscosity not less than 100 centipoises in 2 per cent aqueous solution.

For a cellulose ether having a given ratio of ionic to non-ionic groups the higher the cellulose ether content of the set-retarded calcium sulphate hemihydrate the greater is the retarding effect displayed, but for general purposes the cellulose ether content required will be from 0.1 to 3 per cent of the total weight.

The set-retarded calcium sulphate hemihydrate of the invention may be used in plaster mixtures containing hydrated lime and such mixtures have enhanced water-retaining properties and ease of spread.

The set-retarded calcium sulphate hemihydrate of our invention, when used in plaster mixtures, need not be prepared as a separate mixture. All the ingredients of the

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plaster may, for example, be mixed together so that the cellulose ether is brought into contact with the calcium sulphate hemihydrate within the mixture. It will be obvious that the retarding effect of the cellulose ether will be displayed as effectively in such mixtures as if it were first mixed with the calcium sulphate hemihydrate, and our invention includes the set-retarded calcium sulphate thus prepared.

If desired, other set-retarding ingredients may be incorporated in the calcium sulphate hemihydrate of the invention without detracting from the set-retarding effect of the cellulose ether.

In our co-pending United Kingdom Patent Application No. 13902/63 Serial No. 988,302 there is claimed a dry plaster mixture comprising dry hydrated lime, a retarded calcium sulphate hemihydrate and a small percentage of a finely divided water-soluble cellulose ether containing both ionic and non-ionic etherifying substituent groups in the cellulosic chain, the degree of substitution of the ether by the ionic groups being such that it does not form an insoluble calcium salt in an alkaline solution containing calcium ions, the ratio, by weight, of calcium sulphate hemihydrate to calcium hydroxide being from 4:1 to 1:4, and we make no claim in this specification to any mixture claimed in United Kingdom Patent Application No. 13902/63.

The invention is further illustrated by the following Examples in which all parts are by weight.

#### Example 1

100 parts of a consignment of dry calcium sulphate hemihydrate were mixed with 44 parts water and the time taken to set was found to be 14 minutes. 0.4 parts of methyl sodium carboxymethyl cellulose, having an average degree of substitution of 2.0 methyl groups and 0.15 sodium carboxymethyl groups per anhydroglucose unit and a viscosity of 950 centipoises in 2 per cent aqueous solution, were mixed with 100 parts of the calcium sulphate hemihydrate and 44 parts water were added to form a paste. The setting time of the paste was 37 minutes.

#### Example 2

A dry plaster mixture was prepared by mixing together 100 parts of calcium sulphate hemihydrate, 100 parts of hydrated lime powder and 2 parts of the methyl sodium carboxymethyl cellulose used in Example 1. 150 parts water were mixed with the dry mixture to form a paste which had a setting time of 62 minutes which is comparable with the setting time of similar mixtures containing one of the commonly used retarding materials in equivalent proportion. This paste was, immediately after mixing, usable as a finishing plaster. In a test in

which the rate of water loss to an absorptive background was measured, the water loss factor was 0.06 g./min. The setting time of a similar mixture but without methyl sodium carboxymethyl cellulose was 27 minutes and its water loss factor was 0.38 g./min.

#### Example 3

A dry plaster mixture was prepared by mixing together 100 parts of calcium sulphate hemihydrate, 100 parts of hydrated lime powder and 2 parts of methyl sodium carboxymethyl cellulose having an average degree of substitution of 2 methyl groups and 0.25 sodium carboxymethyl groups per anhydroglucose unit and a viscosity of 1,250 centipoises in a 2 per cent aqueous solution. 150 parts of water were mixed with the dry mixture to form a paste which had a setting time of 60 minutes and which, immediately after mixing, was usable as a finishing plaster. In a test in which the rate of water loss to an absorptive background was measured the water loss factor was 0.07 grams per minute. The setting time of a similar mixture but without methyl sodium carboxymethyl cellulose was 27 minutes and the water loss factor was 0.38 grams per minute.

#### Example 4

A dry plaster mixture was prepared by mixing together 100 parts of calcium sulphate hemihydrate, 100 parts of hydrated lime powder and 2 parts of methyl sodium carboxymethyl cellulose having an average degree of substitution of 2 methyl groups and 0.75 sodium carboxymethyl groups per anhydroglucose unit and a viscosity of 100 centipoises in 2 per cent aqueous solution. 150 parts of water were mixed with the dry mixture to form a paste which had a setting time of 120 minutes and which, immediately after mixing, was usable as a finishing plaster. The water loss factor, determined as in Example 4, was 0.07 grams per minute. The setting time of a similar mixture but without methyl sodium carboxymethyl cellulose was 20 minutes and the water loss factor was 0.38 grams per minute.

#### Example 5

A dry plaster mixture was prepared by mixing together 100 parts of calcium sulphate hemihydrate, 50 parts of hydrated lime, 50 parts of ground limestone and 1.6 parts of methyl sodium carboxymethyl cellulose having an average degree of substitution of 1.8 methyl groups and 0.15 sodium carboxymethyl groups per anhydroglucose unit and a viscosity of 1000 centipoises in 2 per cent aqueous solution. On mixing with 120 parts water the resultant plaster was found to have a setting time of 32 minutes and a water loss factor (determined as in Example 1) of 0.06 grams per minute. The setting time of an equivalent mix from

which the methyl sodium carboxymethyl cellulose had been omitted was 13 minutes and the water loss factor was 0.47 grams per minute.

5 WHAT WE CLAIM IS:—

1. Set-retarded calcium sulphate hemi-  
hydrate comprising, as set-retardant, a  
water-soluble cellulose ether containing both  
ionic and non-ionic etherifying substituent  
10 groups in the cellulose chain, the degree of  
substitution of the ether by the ionic group  
being such that the ether does not form an  
insoluble calcium salt in the presence of a  
saturated solution of calcium hydroxide.
- 15 2. Set-retarded calcium sulphate hemi-  
hydrate as claimed in Claim 1 in which the  
cellulose ether has ionic substituent groups  
comprising sodium carboxymethyl groups.
3. Set-retarded calcium sulphate hemi-  
20 hydrate as claimed in Claim 1 or Claim 2 in  
which the cellulose ether has non-ionic sub-  
stituent groups comprising alkyl, hydroxy-  
alkyl, or alkylhydroxyalkyl groups.
4. Set-retarded calcium sulphate hemi-

hydrate as claimed in any of Claims 1 to 3 25  
wherein the cellulose ether is sufficiently  
fine to pass a 50 mesh B.S. sieve.

5. Set-retarded calcium sulphate hemi-  
hydrate wherein the cellulose ether has a  
viscosity not less than 100 centipoises in 2 30  
per cent aqueous solution.

6. Set-retarded calcium sulphate hemi-  
hydrate as claimed in any of Claims 1 to 5  
wherein the cellulose ether constitutes from  
0.1 to 3 per cent of the total weight. 35

7. Dry plaster mixtures comprising a  
set-retarded calcium sulphate hemihydrate  
as claimed in any of Claims 1 to 6.

8. Wet plaster mixtures comprising set-  
retarded calcium sulphate hemihydrate as 40  
claimed in any of Claims 1 to 6 gauged  
with water.

9. Set-retarded calcium sulphate hemi-  
hydrate as claimed in claim 1 substantially 45  
as herein described.

WALTER SCOTT  
Agent for the Applicants.